

What Is Claimed Is:

1. A method of making lithium metal phosphate compound comprising the step of:

reacting a particulate admixture of starting materials in a non-oxidizing atmosphere and at a temperature sufficient to form a lithium transition metal phosphate reaction product, wherein said admixture of starting materials comprises at least one first metal constituent, at least one phosphate compound, at least one lithium compound, and at least one particulate reducing agent.

2. The method of claim 1 wherein the reaction is carried out at a temperature in a range between about 500°C and about 1200°C.

3. The method of claim 1 wherein the at least one phosphate compound is selected from the group consisting of lithium dihydrogen phosphate, diammonium hydrogen phosphate, ammonium dihydrogen phosphate, transition metal phosphates, and mixtures thereof.

4. The method of claim 1 further comprising the step of:

admixing the starting materials with at least one second metal constituent wherein the metal of the second metal constituent differs from the metal in the first metal constituent.

5. The method of claim 4 wherein the at least one second metal constituent is selected from the group consisting of:

transition metals selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, and mixtures thereof;

oxides of transition metals selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, and mixtures thereof;

carbonates of transition metals selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, and mixtures thereof;

phosphates of transition metals selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, and mixtures thereof;

non-transition metals selected from the group consisting of Mg, Ca, Sr, Pb, Sn, Ba, Be, Al, B, and mixtures thereof;

hydroxides of non-transition metals selected from the group consisting of Mg, Ca, Sr, Pb, Sn, Ba, Be, and mixtures thereof;

oxides of non-transition metals selected from the group consisting of Mg, Ca, Sr, Pb, Sn, Ba, Be, and mixtures thereof; and

mixtures thereof.

6. The method of claim 1 wherein the first metal constituent of the particulate starting material is selected from the group consisting of:

transition metals;

oxides of transition metals;

carbonates of transition metals;

phosphates of transition metals; and mixtures thereof, wherein the metal is selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, and mixtures thereof.

7. The method of claim 1 wherein the particulate reducing agent is selected from the group consisting of:

transition metal constituents selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, TiO_2 , and mixtures thereof;

non-transition metals selected from the group consisting of Mg, Ca, Sr, Pb, Sn, Ba, Be, Al, B, and mixtures thereof;

non-metal constituents selected from the group consisting of silicon (Si), silicon oxide (SiO_2), carbon, and mixtures thereof; and

mixtures thereof.

8. The method of claim 1 wherein the at least one lithium compound is lithium fluoride, and the resulting compound is a lithium metal fluorophosphate reaction product having the nominal formula $LiMPO_4F$, where M is a metal selected from the group consisting of iron, cobalt, nickel, copper, chromium, titanium, vanadium, manganese, and mixtures thereof.

9. The method of claim 8 wherein the lithium metal fluorophosphate reaction product is characterized by a triclinic structure.

10. The method of claim 8 wherein the at least one phosphate compound is capable of at least partial reduction and the at least one first metal constituent is selected from the group consisting of:

- transition metals;
- oxides of transition metals;
- phosphates of transition metals;
- carbonates of transition metals; and
- mixtures thereof.

11. The method of claim 8 wherein the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, lithium dihydrogen phosphate, transition metal phosphates, and mixtures thereof.

12. The method of claim 8 wherein the at least one metal constituent is iron oxide, the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, and mixtures thereof, and the resulting reaction product is a lithium iron fluorophosphate represented by the nominal formula LiFePO_4F .

13. The method of claim 8 wherein the at least one metal constituent is chromium oxide, the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, and mixtures thereof, and the resulting reaction product is a lithium chromium fluorophosphate represented by the nominal formula LiCrPO_4F .

14. The method of claim 8 wherein the at least one metal constituent is titanium oxide, the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, and mixtures thereof, and the resulting reaction product is a lithium titanium fluorophosphate represented by the nominal formula LiTiPO_4F .

15. The method of claim 8 wherein the at least one metal constituent is vanadium pentoxide, the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, and mixtures thereof, and the resulting reaction product is a lithium vanadium fluorophosphate represented by the nominal formula LiVPO_4F .

16. The method of claim 8 wherein the at least one metal constituent is manganese oxide, the at least one phosphate compound is selected from the group consisting of diammonium hydrogen phosphate, ammonium dihydrogen phosphate, and mixtures, and the resulting reaction product is a lithium manganese fluorophosphate represented by the nominal formula LiMnPO_4F .

17. The method of claim 1 wherein at least one fluoride compound is admixed with the at least one lithium compound, the at least one first metal constituent, and the at least one phosphate compound under conditions such that the resulting compound is a

lithium metal fluorophosphate reaction product having the nominal formula LiMPO_4F , where M is a metal selected from the group consisting of iron, cobalt, nickel, copper, chromium, titanium, vanadium, manganese, and mixtures thereof.

18. The method of claim 1 wherein the source of lithium ions is a compound selected from the group consisting of lithium fluoride, lithium dihydrogen phosphate, lithium carbonate, and mixtures thereof.

19. A method of making a lithium metal fluorophosphate compound comprising the steps of:
mixing starting materials in particle form, comprising at least one metal constituent, a lithium compound, a fluoride compound, and a phosphate compound; and
heating the starting material mixture to a temperature sufficient to form a lithium metal fluorophosphate reaction product comprising lithium, said reduced metal ion, phosphate, and fluoride.

20. A method of making a lithium mixed metal fluorophosphate compound comprising the steps of:
mixing starting materials in particle form, comprising a first metal constituent, a second metal constituent, and at least one phosphate compound;
heating the starting material mixture with a reducing agent in a non-oxidizing atmosphere to a temperature sufficient to form a mixed metal phosphate reaction product comprising a first metal phosphate, and a second metal phosphate;

mixing, in particle form, said first metal phosphate reaction product with a lithium compound and a fluoride compound; and

heating the resulting mixture to a temperature sufficient to form a lithium mixed metal fluorophosphate reaction product, the lithium mixed metal fluorophosphate reaction product comprising the first metal, the second metal, phosphate, fluoride, and lithium.

21. A method of making a metal phosphate compound comprising the steps of:

mixing starting materials in particle form, the starting materials including at least one metal constituent, and at least one phosphate compound; and

heating the starting material mixture with a reducing agent in a non-oxidizing atmosphere to a temperature sufficient to form a metal phosphate reaction product comprising a metal and phosphate anion.

22. The method of claim 21 further comprising:
mixing said metal phosphate in particulate form with a particulate lithium compound; and

heating the ensuing mixture to a temperature sufficient to form a lithium metal phosphate compound, the lithium metal phosphate compound comprising a metal, a phosphate, and a lithium.

23. The method of claim 21 further comprising:
mixing said metal phosphate in particulate form with a particulate lithium compound, and a particulate fluoride compound; and

heating the ensuing mixture to a temperature sufficient to form a lithium metal fluorophosphate reaction product, the lithium metal fluorophosphate comprising a metal, a phosphate, a fluoride and a lithium.

24. The method of claim 21 further comprising: mixing said metal phosphate in particulate form with lithium fluoride; and

heating the ensuing mixture to a temperature sufficient to form a lithium metal fluorophosphate reaction product, the lithium metal fluorophosphate comprising a metal, a phosphate, a fluoride, and a lithium.

25. The method of claims 21, 22, 23, or 24 wherein said metal constituent is a compound of a metal selected from the group consisting of Fe, Co, Mn, V, Ti, Cr, Ni, Cu, and mixtures thereof.

26. The method of claim 24 where said metal constituent comprises iron oxide;

said one phosphate compound comprises diammonium hydrogen phosphate, or ammonium dihydrogen phosphate;

said metal phosphate reaction product comprises iron phosphate;

said lithium compound comprises lithium fluoride; and

said lithium metal fluorophosphate reaction product comprises lithium iron fluorophosphate represented by the nominal formula LiFePO_4F .

27. The method of claim 24 wherein
said metal constituent comprises chromium
oxide;

 said one phosphate compound comprises
diammonium hydrogen phosphate, or ammonium dihydrogen
phosphate;

 said metal phosphate reaction product comprises
chromium phosphate;

 said lithium compound comprises lithium
fluoride; and

 said lithium metal fluorophosphate reaction product
comprises lithium chromium fluorophosphate represented by
the nominal formula LiCrPO_4F .

28. The method of claim 24 where
said metal constituent comprises titanium
oxide;

 said one phosphate compound comprises
diammonium hydrogen phosphate, or ammonium dihydrogen
phosphate;

 said metal phosphate reaction product comprises
titanium phosphate;

 said lithium compound comprises lithium
fluoride; and

 said lithium metal fluorophosphate reaction
product comprises lithium titanium fluorophosphate
represented by the nominal formula LiTiPO_4F .

29. The method of claim 24 where
said metal constituent comprises vanadium
pentoxide;

said one phosphate compound comprises diammonium hydrogen phosphate, or ammonium dihydrogen phosphate;

said metal phosphate reaction product comprises vanadium phosphate;

said lithium compound comprises lithium fluoride; and

said lithium metal fluorophosphate reaction product comprises lithium vanadium fluorophosphate represented by the nominal formula LiVPO_4F .

30. The method of claim 24 where said metal constituent comprises manganese oxide;

said one phosphate compound comprises diammonium hydrogen phosphate, or ammonium dihydrogen phosphate;

said metal phosphate reaction product comprises manganese phosphate;

said lithium compound comprises lithium fluoride; and

said lithium metal fluorophosphate reaction product comprises lithium manganese fluorophosphate represented by the nominal formula LiMnPO_4F .

31. The method of claim 24 wherein said reducing agent is selected from the group consisting of: transition metals selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, Zn, Cd, and mixtures thereof;

non-transition metals selected from the group consisting of Mg, Ca, Sr, Pb, Sn, Ba, Be, Al, B, and mixtures thereof;

non-metal constituents selected from the group consisting of silicon (Si), silicon oxide (SiO), carbon, and mixtures thereof; and
mixtures thereof.

32. A method of making a lithium transition metal oxide compound for use as a cathode active material comprising the steps of:

admixing starting materials in particle form, including at least one lithium compound, at least one transition metal oxide compound, and at least one particulate reducing agent; and

heating the starting material mixture in a non-oxidizing atmosphere to a temperature sufficient to form a lithium transition metal oxide reaction product.

33. The method of claim 32 wherein the metal in the at least one transition metal oxide is selected from the group consisting of V, Fe, Mn, Cr, Cu, and mixtures thereof.

34. The method of claim 32 wherein the particulate starting material further includes at least one second metal constituent from the group consisting of Fe, Mn, V, Cr, Cu, and mixtures thereof.

35. The method of claim 32 wherein the particulate reducing agent is selected from the group consisting of:

transition metal constituents selected from the group consisting of Fe, Co, Ni, Mn, Cu, V, Ti, Cr, TiO, and mixtures thereof;

non-transition metals selected from the group consisting of Mg, Ca, Zn, Sr, Pb, Cd, Sn, Ba, Be, Al, B, and mixtures thereof;

non-metal constituents selected from the group consisting of silicon (Si), silicon oxide (SiO), carbon, and mixtures thereof; and

mixtures thereof.

36. The method of claim 3 wherein the transition metal phosphate is selected from the group consisting of $Mn_3(PO_4)_2$, $FePO_4$, $Fe_3(PO_4)_2$, $Zn_3(PO_4)_2$, $TiPO_4$, $CrPO_4$, $Mg_3(PO_4)_2$, and mixtures thereof.